

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.:	10/797,938	Confirmation No.:	4873
Applicant(s):	Douglas R, Svenson, Jian Li		
Filed:	03/11/2004		
Art Unit:	1623		
Examiner:	White, Everett		
Title:	PROCESS FOR MANUFACTURING HIGH PURITY XYLOSE		

Docket No.: 046088/267693  
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Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

October 31, 2007

DECLARATION OF DR. JIAN LI UNDER 37 C.F.R. § 1.132

Sir:

I, Jian Li, hereby declare and state that:

1. I am one of the inventors of the claimed invention of the above-identified U.S. Patent Application Serial No. 10/797,938, titled "Process for Manufacturing High Purity Xylose" (hereinafter referred to as "the Application"). I am currently employed by Rayonier, Inc. the assignee of the above-identified application, and have been at all times during and following the invention described by the Application.

2. I have studied and worked in the area of paper and pulp processes including pulp and fiber strength and have considerable experience in this field. Since December 2000 to the present time, I have been conducting research on the in-line control of the kraft pulping kinetics, the physical chemistry of the alkaline hemicellulose solutions, and separation technologies of aqueous solution at

Rayonier Research Center. I obtained a Ph.D. degree in Chemical Engineering from McGill University, Montreal in December 1989; a Master of Engineering degree in Chemical Engineering from McGill University; and a Bachelor of Engineering degree from Tianjin Institute of Light Industry, China. From 1990 to 1997, I was employed with Pulp and Paper Research Institute of Canada (PAPRICAN), first as Assistant Scientist, then Associate Scientist, and eventually was promoted to Scientist. In December 1997, I joined the Institute of Paper Science & Technology in Atlanta, GA as an Associate Professor of Chemical Engineering where I conducted research and teaching until December 2000. A significant amount of my research during the 10 years I was at PAPRICAN and the Institute of Paper Science and Technology was directed towards the effect of hemicellulose change due to pulping, bleaching, yield improvement on paper pulp fiber physical strength, membrane separation of macromolecules in pulp industrial effluent, and fundamental transport phenomena in membrane separation. I have published numerous articles in some of the most prestigious scientific journals in this research field, including *Journal of Pulp & Paper Science*; *TAPPI Journal*; *The Canadian Journal of Chemical Engineering*; *Chemical Engineering Science*, *Environmental Science & Technology*; and *Industrial Engineering Chemistry Research*. I have also presented conference papers in many of the most significant conferences in the Chemical Engineering and Pulp & Paper Science field, including the International Symposium on Wood and Pulping Chemistry, the CPPA Annual Conference and the TAPPI Pulping Conference. I am also a coinventor in 5 U.S. patents. A list of the various publications that I have authored or have coauthored is provided in the appendix.

3. I have reviewed the Office Actions dated January 12, 2007 and June 22, 2007, including the basis of rejection of Claims 1 – 48 as being obvious over a combination of US Patent No. 6,512,110 to Heikkila et al. and U.S. Patent No. 3,988,198 to Wilson et al.

4. In the Office Action of June 22, 2007, the Examiner asserts that the claims do not distinguish between paper grade pulp and dissolving grade pulp. This statement is incorrect. In the art of pulp technology and related processes, it is commonly understood and recognized that a pre-hydrolyzed pulp is a dissolving grade pulp. In Applicants' Response date April 12, 2007, Applicants specifically cited references that establish that a pre-hydrolyzed pulp is a dissolving grade pulp. See for example, Gary A. Smook, HANDBOOK FOR PULP & PAPER TECHNOLOGIST, 2d, page 79 (1999) (hereinafter ("Smook")), that discusses that the prehydrolysis step is used to make a dissolving-grade pulp; and Ingruber et al, Editor, Vol. 4 Sulfite SCIENCE & TECHNOLOGY, PULP AND PAPER MANUFACTURE, 3d ed., pg. 233, that describes that prehydrolysis is a step used in the kraft process that is necessary to make dissolving type pulps. Thus, in the pulp industry, the term prehydrolyzed pulp has a specific meaning that refers to the process used to make a dissolving grade pulp.

5. I disagree with the assertions set forth in the Office Action dated January 12, 2007 that it would be obvious to one of ordinary skill in the art to substitute the pulp used in the process for the production of xylose from hardwood pulp of the Heikkila patent with the prehydrolyzed pulp of Wilson. In particular, as a person having a high level of skill in the art to which these patents pertain, I believe that the Examiner's rationale for asserting that it would be obvious to use Wilson's pulp in the method/system of Heikkila is based on an incorrect or incomplete understanding of the teachings of these references, and is therefore incorrect.

6. Heikkila is concerned with a process for the preparation of xylose from a paper-grade hardwood pulp which includes treating the pulp with a xylanase enzyme treatment. See Abstract. Heikkila also repeatedly emphasizes that the

process is directed to extraction of xylose from paper-grade pulp. See for example, Abstract; Column 1, line 8 – 12; column 5, lines 14 – 16; Examples 1 – 21 and the Claims. In particular, Heikkila emphasizes that the xylanase treatment “improves the quality of the pulp for use as a source of xylan in that it improves the solubility of xylan whereby xylan is more easily removed from pulp. This results in increased recovery of xylan and, accordingly, higher yields of xylose. Without being bound to the theory, these improvements are believed to have their basis in the increased solubility of xylan, which is caused by the xylanase treatment.” See column 5, lines 5 – 13. A further objective of Heikkila’s process is the production of a dissolving grade pulp. For example, Heikkila states “the xylanase treatment results in a simultaneous production of dissolving-grade pulp of very high quality and high yields of xylose.” See column 5, lines 43 – 49, see also Column 9, lines 15 – 19.

7.     Wilson on the other hand is directed to a completely different and unrelated process than that of Heikkila. Specifically, Wilson is directed to a process of recovering and reutilizing pulping chemicals from waste effluents produced during the manufacture of dissolving grade pulps. See column 1, lines 9 – 12. Wilson teaches that heat treatment of spent effluent renders the “effluents suitable for use in cooking, bleaching, and refining of dissolving pulps without adverse effect on the quality of the pulps.” See column 2, lines 23 – 28. According to the teachings of Wilson, heat treatment of the spent effluent stream causes thermal degradation of the hemicelluloses that are dissolved in the spent effluent “so that hemicelluloses will no longer precipitate out on cellulosic materials or otherwise interfere with the action of the caustic soda when the effluents are utilized for treating cellulosic material.” See column 2, lines 66 – column 3, line 1.


8. One of ordinary skill in the art would not be motivated to combine Heikkila with Wilson. First, Heikkila is directed to an entirely different field of application from the objective and purpose of Wilson. As noted in paragraph 5, Heikkila is directed to a process of recovering xylose from paper grade. This is quite different from the problem addressed by Wilson, namely the recovery of spent chemicals from caustic waste effluent. In fact, Wilson is completely silent with respect to the recovery of xylose. Because of the significant differences to which these patents are directed, a person of ordinary skill in the art would not consider using the prehydrolyzed pulp of Wilson in the xylanase enzyme treatment process of Heikkila.

8. Second, one of the preferred objectives of the process of Heikkila is to produce a dissolving grade pulp. That is, the end product of Heikkila's process is the simultaneous production of both xylose and a dissolving grade pulp. As such, one of ordinary skill in the art would not select the use of a dissolving grade pulp (i.e., the prehydrolyzed pulp of Wilson) as a starting product in the process of Heikkila. It would serve no purpose to modify the Heikkila process to include a desired end product as a starting product. Accordingly, one of skill in the art would not be motivated to modify the process of Heikkila to substitute a paper grade pulp with the prehydrolyzed pulp of Wilson.

9. Third, substituting a dissolving grade pulp (e.g., Wilson's prehydrolyzed pulp) for a paper grade pulp in the process of Heikkila would result in significant loss of pulp viscosity, which would render the resulting pulp unusable. The pulp viscosity, or IV, is a measure of the cellulose chain length, the higher IV, the longer the chain. For paper grade pulp, since it contains 15 -20% short chain hemicellulose, the IV value of the pulp is a measure of the average of the mixture of cellulose and hemicellulose. Accordingly, when hemicellulose is removed from the pulp without degrading the cellulose, the resulting pulp will have a

higher IV value than the starting pulp. In the case of the paper grade pulp used in the examples of Tables I – III, the cellulose chain should be greater than what is expected based on an IV of 7.97 (see column 8, line 41). As such, when hemicellulose is removed from the pulp without degrading the cellulose, it would be expected that the resulting pulp would have a higher IV because of the increase in cellulose concentration. However, based on the values shown in Tables I – III of Heikkila, the IVs of the treated pulp were predominately lower, and in some cases significantly lower, mostly because of the repeat treatment by the strong warm caustic and enzyme. This IV loss is probably fine when starting with a paper grade pulp with high IVs. However, a prehydrolyzed pulp after cooking has a relatively low IV already, generally below 7.0. As such, if a prehydrolyzed kraft pulp, such as the one described in Wilson, is treated in accordance with Heikkila's process, the resulting pulp would have an IV that would be too low to be used as a dissolving grade pulp. Accordingly, one of ordinary skill in the art would avoid using Wilson's prehydrolyzed pulp in the process of Heikkila.

10. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.



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Jian Li Ph.D

## Appendix

### List of publications authored by Dr. Li

#### 1. Journal Publications

J. Samps, J. Li, "How does mass transfer affect the effectiveness of AQ", **Appita J.**, Vol 57 No 2, p.132-136, 2004.

C. Luthe, R. Berry, J. Li, "Polysulfide for yield enhancement in sawdust pulping: Does it work?" **Pulp & Paper Canada**, p. 32-37, 105(1), 2004

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J.Y. Zhu, X-S. Chai, X.J. Pan, Q. Luo, and J. Li, Quantification and Reduction of Organic Sulfur Compound Formation in a Commercial Wood Pulping Process, **Environ Sci. & Techno.**, 36(10), 2269-2272, 2002.

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K. Goel, T. Radiotis, R. Eisner, G. Sherson, J. Li, "Switchgrass: a potential pulp fibre source," **Pulp & Paper Canada**, 101:6, p.41-45, 2000.

J. Li, G. Moeser, L. Roen, "Nonuniformity of carbohydrate degradation during kraft pulping - measurement and modeling using a modified G-factor," **Ind. Eng. Chem. Res.**, vol.39, no. 4, p.916-921, 2000.

J. Li, M. MacLeod, R. Berry, J. McPhee, "Improving extended delignification technology for kraft pulping, Part II: Co-current two-stage continuous cooking ", **Pulp and Paper Canada**, vol. 100, No. 11, p.48, 1999.

J. Li, C. Mui, "Effect of lignin diffusion on kraft delignification kinetics as determined by liquor analysis. Part I: An experimental study", **J. Pulp & Paper Sci.**, vol.25, No.11, p.373, 1999.

T. Radiotis, J. Li, K. Goel, R. Eisner, "Fiber characteristics, pulpability, and bleachability of switchgrass," **Tappi J.**, vol. 82, No. 7, p.100, 1999.

Z. Li, H. Ma, G. Kubes, J. Li, "Synergistic Effect of Kraft Pulping with Polysulphide and Anthraquinone on Pulp Yield Improvement", **J. Pulp & Paper Sci.**, vol. 24, No.8, p.237, 1998.

J. Li, A. Phenix, M. MacLeod, "Diffusion lignin macromolecules within the fibre walls of kraft pulp. Part I: Determination of the restricted diffusion coefficient under alkaline conditions", **Canadian J. Chemical Engineering**, vol. 75, No.1, p.16, 1997.

J. Li, M.G. Paice, J.M. MacLeod, L. Jurasek, "The bleachability improvement of kraft pulp by alkaline leaching and xylanase treatment - their industrial implications", **J. Pulp & Paper Sci.**, vol.22, No. 6, p.J207, 1996.

J. Li, T. O'Hagan, M. MacLeod, "Using ultrafiltration to analyze the molecular weight distribution of kraft lignin at pH 13", **Canadian J. Chemical Engineering**, vol.74, Feb., p.110, 1996.

J. Li and A.R.P. van Heiningen, "Kinetics of solid state reduction of sodium sulfate by carbon", **J. Pulp & Paper Sci.**, vol.21, No. 5, p.J165, 1995.

J. Li, and A.R.P. van Heiningen, "The rate process of H<sub>2</sub>S emission during steam gasification of black liquor char", **Chemical Engineering Science**, vol. 49, No. 24, p.4143, 1994.

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J. Li and A.R.P. van Heiningen, "The effect of sodium catalyst dispersion on the CO<sub>2</sub> gasification rate". **MRS Symposium series**, vol.111, MRS, p.441,1988.

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## **2. Patents**

J. Li, S.F. Boller, "Celluloseic fiber pulp and highly porous paper products produced therefrom", **US Patent**, No. 7,285,184, Oct. 23, 2007.

O. Ali, J. Cenicola, J. Li, J. Taylor, "Process for producing alkaline treated cellulosic fibers", **US Patent**, No. 6,896,810, May 24, 2005.

C. Luthé, J. Li, R. Berry, "Improved Pulping Process", **US Patent**, No. 6,153,052, Nov. 28, 2000.

J. Li, "Two stage kraft cooking", **US Patent**, No. 5,522,958, June 4, 1996.

A.R.P. van Heiningen, J. Li, and J. Fallavollita, "Low temperature recovery of kraft

black liquor", **US Patent, No. 5,174,860**, Dec. 29, 1992. (= **Can. Patent, No. 1,313,577**, Feb. 1993.).

O. Ali, J. Li, S. Rogers, "Method of concentrating pulp mill extracts", **US patent application, No. 20,060,016,751**, Jan. 26, 2006

J. Li, X.S. Chai, J. Zhu, "Simultaneous and Rapid Determination of Effective Alkali, Carbonate and Sulfide Concentrations in Kraft Liquors", **US patent Application, No. 20,050,139,337**, June 30, 2005.

### **3. Representative Conference Publications**

Li, J.; Moeser, G.; Rosen, L., "Non-uniformity of carbohydrate degradation during kraft pulping - measurement and modeling using a modified "G" factor," **the 10<sup>th</sup> International Symposium on Wood and Pulp Chemistry**, Yokohama, Japan, June 7-10, p.400, 1999.

Chai, X-S.; Zhu, J.; Li, J., "A simple and rapid method for determination of hexeneuronic acid." **the 10<sup>th</sup> International Symposium on Wood and Pulp Chemistry**, Yokohama, Japan, June 7-10, p.390, 1999.

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Goel, K.; Eisner, R.; Sherson, G.; Radiotis, T.; Li, J., "Switchgrass: Potential Pulp-Fibre Source", **Proceeding of 84<sup>th</sup> CPPA annual conference**, Montreal, B109-114 (January 30, 1998).

In re:       Jian Li, Douglas R, Svenson  
Appl. No.:   10/797,938  
Filed:       03/11/2004  
Page 11 of 11

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J. Li, M. MacLeod, J. McPhee, R. Berry, T. O'Hagan, B. Thomas, B. Tristram, R. Kowalska "Improving extended delignification technology for kraft pulping, Part I: Papri-Batch Cooking", **Proceeding of TAPPI pulping conference**, San Francisco, Oct., p.577(1997).